



Permeability Testing of Impacted Composite Laminates for Use on Reusable Launch Vehicles

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TECHNICAL MEMORANDUM

PERMEABILITY TESTING OF IMPACTED COMPOSITE LAMINATES FOR USE ON REUSABLE LAUNCH VEHICLES

1. INTRODUCTION

As composite laminates are being considered for use in liquid propulsion systems, microcracking due to foreign object impact damage becomes very important, especially if a tank or feedline is to be unlined. If a component that carries liquid or gaseous hydrogen develops an area of microcracking, hydrogen can leak out of the component and pose a serious threat to the vehicle. Since it has been shown that nonvisible impact damage can cause a composite feedline to leak,¹ a better understanding of the material's resistance to microcracking is needed. Microcracking can also occur due to thermal and mechanical stresses and fatigue; however, this study will deal only with foreign object impact damage, a very real threat to all composite parts. The most quantifiable way of determining how much leakage may occur after an impact event is to test the material for permeability. Permeability testing has been used in the past on composites to determine the porosity of rocket nozzle material.² Fluid permeability has been tested on some composite structures to be used as fuel tanks.^{3,4} ASTM D1434, *Standard Test Method for Determining Gas Permeability Characteristics of Plastic Film and Sheeting*, exists for gas permeability testing of plastic film and sheeting, and it is from this test methodology that the one in this study was adapted.

For this study, prepreg composed of IM7 fiber in a five-harness weave architecture impregnated with Bryte Technologies EX1522 epoxy resin was used to construct four-ply laminates with the layup sequence (0/90,0/90)_s. These specimens were representative of one of the carbon/epoxy systems being evaluated for use in constructing feedlines for future launch vehicles. A drop-weight impact tester with a 0.25-in.-diameter tup was used to impart varying levels of damage from almost nondetectable to near penetration. The specimens were then secured in a fixture that could supply a positive pressure of helium (He) gas on one side and allow a leak detection solution to be applied to the other side. This gave a qualitative assessment of permeability-after-impact. The specimens were then secured in an apparatus that could give quantitative results. Since this testing technology is new, a great deal of test verification was performed and will be presented in this study.

2. EXPERIMENTAL PROCEDURES

This section will explain how the impact and subsequent permeability testing were performed. Results will be given in section 3.

2.1 Specimens

The specimens were manufactured from carbon/epoxy prepreg, which was in a five-harness satin weave form. A 36×24 in. panel was laid up in a bidirectional configuration on a flat aluminum tool. This gave the laminate a layup sequence of $(0/90, 0/90)_s$. The panel was then vacuum bagged and autoclave cured according to the manufacturer's recommended cure cycle. The cured laminate was then cut into 3×3 in. specimens. The nominal thickness of the specimens was 10 mils.

2.2 Impact Testing

The 3-in.-square specimens were impacted at various levels using a drop-weight apparatus. The impactor consisted of a 0.25 in. semispherically ended tup that was attached to a dynamic load cell to gather instrumented impact data, should it be needed for future analysis. (None of the instrumented data were used in the study presented in this paper). The falling mass had a total weight of 2.51 lb and drop heights of 4, 6, 8, 10, and 12 in. were used. The specimen was simply supported over a 2-in.-square opening. A schematic of the impact setup is given in figure 1. Two specimens were impacted from each drop height to give a total of 10 impacted specimens in order to check for repeatability of results. After each specimen was impacted, images of both the front and back surface damage were recorded with a digital camera at a magnification of approximately $\times 5$.

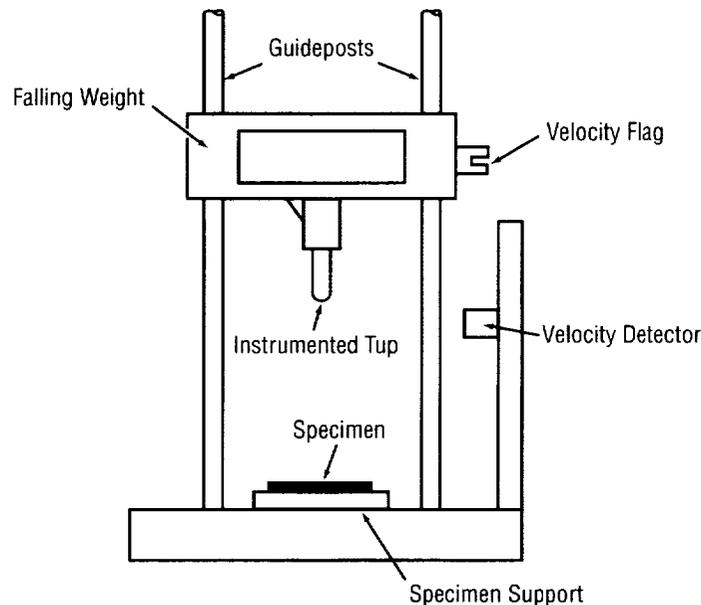


Figure 1. Schematic of impact apparatus.

2.3 Leak Check

After all specimens had been impacted and the surface damage recorded, the specimens were mounted in an apparatus to check for leakage of helium gas when a positive pressure was applied to one side. A “bubble-type” leak detector solution was used, and a digital image was made of the leaks for each specimen. A schematic of the apparatus used to check for leaks is shown in figure 2. A sample image of a leak is given in figure 3.

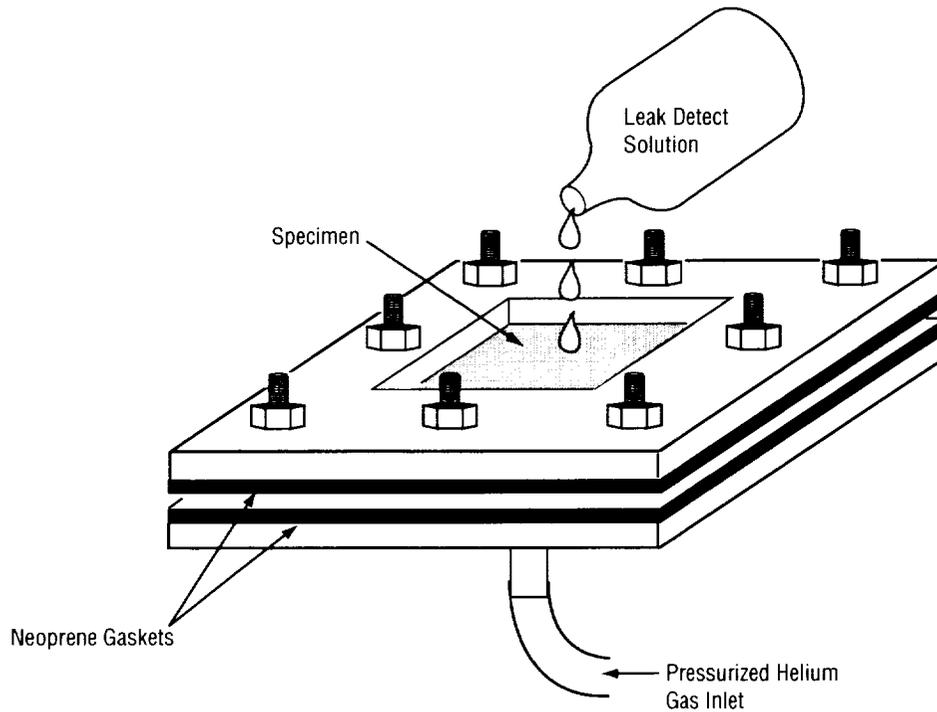


Figure 2. Schematic of leak detection apparatus.

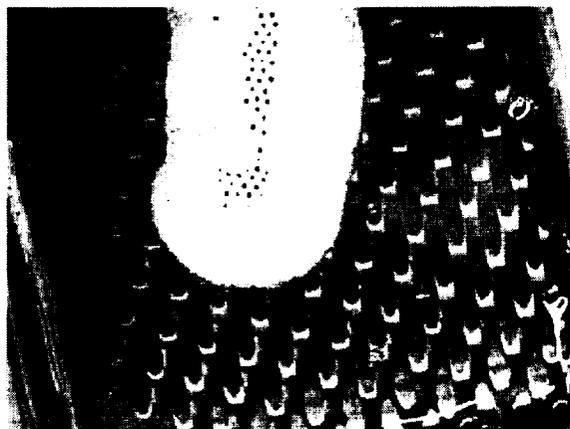


Figure 3. Specimen displaying a leak using a bubble-type leak detection fluid.

2.4 Permeability Testing

After the specimens had thoroughly dried from the leak detection procedure, they were ready for permeability testing. This section will be devoted to a discussion of the apparatus used and its limitations and verification.

2.4.1 Permeability Apparatus

The apparatus used in this study is based loosely on ASTM D1434, using the volumetric technique. This apparatus could have been used, but modifications would have to be made for the larger specimen size needed (to include all the impact damage) and a much more simple apparatus could be used since relatively high rates of permeance were being measured. The apparatus basically consisted of a specimen holder that isolated the two surfaces and allowed a positive pressure of gas to be applied to one side and the gas that leaked through the specimen to escape out the other side. The escaping gas rate was measured via a volumetric displacement method using a liquid “slug” in glass tubes of various diameters. The rate-of-volume change was measured with a stopwatch so the amount of gas escaping through the specimen could be calculated in volume-per-unit time.

It should be noted at this point that, strictly speaking, the term “permeability” can only apply to homogeneous materials and is defined as the “permeance” times the specimen thickness. Permeance is defined as the transmission rate per applied pressure of gas, which is what is being measured in this paper. However, the term “permeability” has been used in the composites industry much more than “permeance” to indicate a transmission rate, and it will be used in this paper in order to avoid confusion.

A sketch of the apparatus is given in figure 4 to aid in the discussion that follows. A test or “run” was typically conducted in the following manner.

The 3 × 3 in. square composite specimen was centered and sandwiched between the top and bottom aluminum plates. A small amount of vacuum grease was placed around the edges of the specimen to ensure a good seal between the specimen and the neoprene gaskets. The eight bolts and nuts were then used to secure the specimen between the two plates. Each bolt had 100 in.-lb of torque applied. The specimen holder was then secured to a cantilever beam above a laboratory table with a C-clamp. An inlet hose from a helium supply tank was then inserted over the bottom nipple to supply the positive pressure to the specimen. A precision pressure gauge was included in the supply line so an accurate reading of the amount of pressure being applied to one side of the specimen could be determined. Typical values of applied pressure ranged from 3 to 50 psi. A liquid “slug” or “blank” was then introduced into the glass tube. Alcohol was usually used as the liquid and enough to introduce a slug length of approximately 3–10 in. was inserted into the top of the tube. The alcohol was then allowed to fall into the bottom U-shape of the Tygon[®] tubing that was snugly attached to the glass tube. The other end of the tubing was then attached to the upper nipple of the specimen holder to channel all leaking gas into the tubing and force the slug into and up the glass. The slug was allowed to reach steady state (constant velocity) and then the time needed to travel a given distance was recorded. This procedure was repeated for various applied pressures of helium so a pressure versus flow rate chart could be created. Section 2.4.2 will present equations and experimental results to show how various parameters could affect the flow rate data.

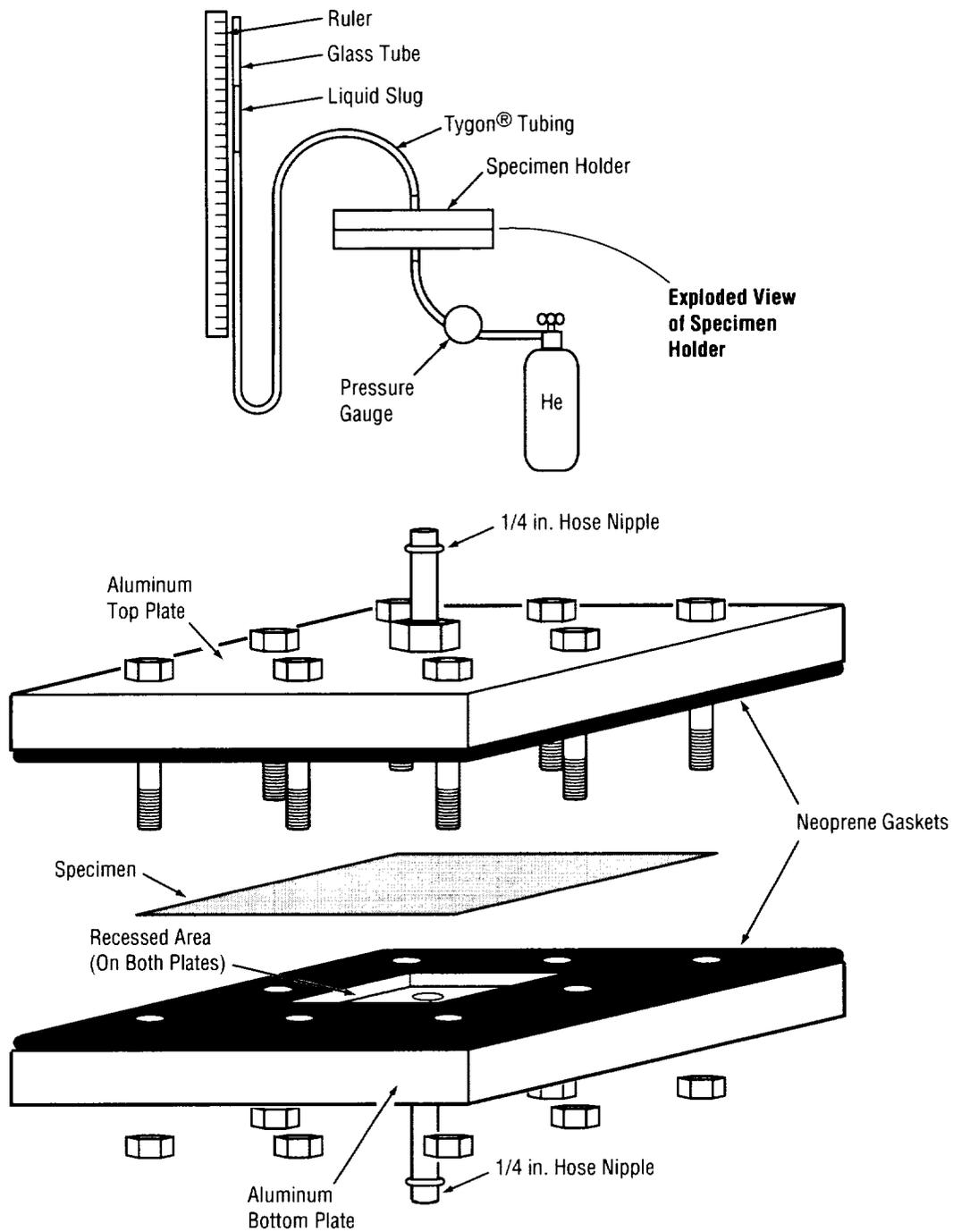


Figure 4. Sketch of permeability apparatus.

2.4.2 Analysis of Apparatus

From past experience and intuitive deductions, it was determined that certain key variables in performing the test may affect the outcome of the flow rate data. These variables were examined using equations of laminar flow to see what variables would most likely affect the data. Figure 5 will be useful in the following discussions.

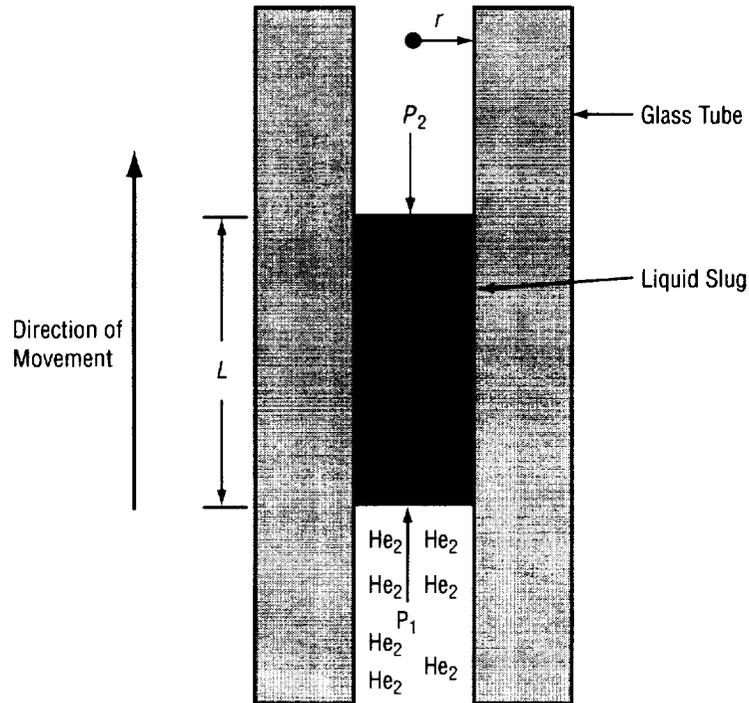


Figure 5. Schematic of liquid slug in glass tube.

Since the end of the tube is unrestricted, p_2 will always equal atmospheric pressure p_a . At the beginning of the experiment, the volume between the top of the specimen and the slug is also at atmospheric pressure (i.e., p_1 also equals p_a). Thus the additional force needed to keep the slug in the tube from falling back down is given by

$$F = \pi r^2 L \rho g \quad (1)$$

where

- r = inner radius of glass tube
- L = length of liquid slug
- ρ = density of slug liquid
- g = acceleration due to gravity.

Thus the additional pressure needed is $F/\text{area} = F/\pi r^2$, or

$$p_g = L \rho g \quad (2)$$

This equation indicates that the pressure needed to keep the slug from falling back down the tube is proportional to the slug length and the liquid's density. Thus to minimize this term, a short slug length and a low density liquid are desired. It should be noted that if the tube is slanted toward the horizontal position, less of the slug liquid will be acting in a downward manner and L will be lessened by the amount $\sin\theta$ where θ is the angle the glass tube makes with the horizontal position.

Assuming the slug to be alcohol, the pressure needed to keep the slug from falling back down is given by

$$p_g = 0.0289(L) \quad , \quad (3)$$

where L is slug length in inches. Thus assuming a 3-in. slug length, p_g will be on the order of 0.086 psi, making $p_1 \approx 14.786$ psi.

Once the slug is moving at a constant velocity up the tube (steady state), the viscous forces of the liquid need to be overcome. This force is given by

$$F = 8\pi\eta Lv \quad , \quad (4)$$

where

- η = viscosity of liquid used for slug
- L = length of slug
- v = velocity of slug traveling up the glass tube.

Thus the additional pressure needed to overcome the viscous forces of the slug is $F/\pi r^2$, or

$$p_\eta = \frac{8\eta Lv}{r^2} \quad . \quad (5)$$

This equation indicates that the pressure needed to overcome the viscous forces of the slug are proportional to the liquid's viscosity, slug length, and velocity, and inversely proportional to the square of the radius of the tube. Thus to minimize this term, a low viscosity liquid, a short slug length, a low slug velocity, and especially a large tube radius are all desired.

The helium leaking through the specimen and filling the volume of the permeability apparatus provides the force (or pressure) necessary to overcome the viscous forces induced by the liquid slug. If this pressure is not large compared to the pressure needed to overcome the viscous forces, then values of flow rate measured will be too small.

As a check for possible errors introduced by these forces, some experiments were carried out varying the angle of the tube, the liquid in the tube, the length of the liquid slug, and the tube size. The results of these experiments follow.

2.4.2.1 Vertical Versus Slanted Glass Tube. It was determined early that if the glass tube could be laid down at a slant, rather than being vertical, the testing would be much simpler. Thus a series of tests was performed with the glass tube in a vertical position and the glass tube laid down at an angle of 5 deg. Specimen 6B was used for all the tests and alcohol was used as the liquid for the slug. Excellent repetition of data was obtained and typical results from a vertical versus a slant tube are shown in figure 6. Since no differences were found, all subsequent tests were performed with the glass tube placed on a wooden board angled at 5 deg.

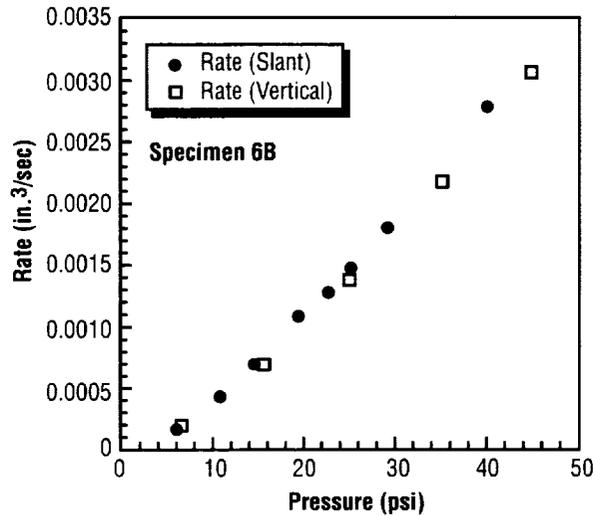


Figure 6. Flow rate results of vertical and slanted glass tube.

2.4.2.2 Type of Liquid Used as a Slug. Alcohol was typically used as the liquid for the slug, but the use of water and alcohol with PhotoFlo[®] to reduce surface tension was examined. Again, specimen 6B was used for these tests and the blank length kept at ≈ 3 in. Results are shown in figure 7. Since no difference was found for the liquid used as the slug, alcohol was chosen for subsequent testing due to its ease of cleaning and fast evaporation rate.

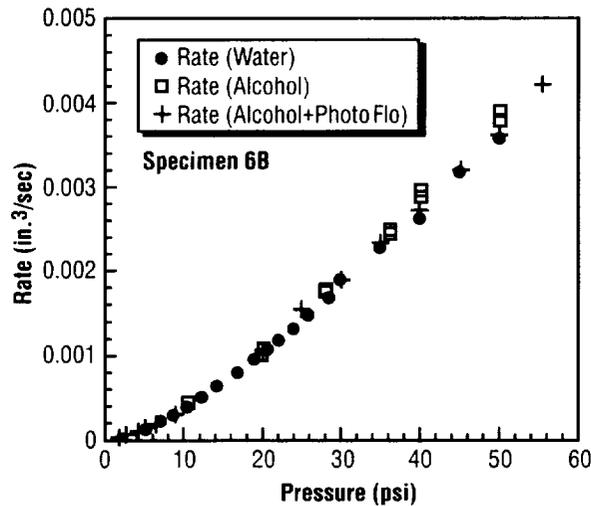


Figure 7. Flow rates for specimen 6B using various liquids for slugs.

2.4.2.3 Length of Slug. It is extremely difficult to obtain a consistent slug length when placing the liquid in the glass tube. Thus a series of tests was performed to see if the slug length affected the measured flow rate. Specimen 5A was used for these data. The results are shown in figure 8. No difference in measured rates was noted, so slug length was not a factor and any convenient length could be used.

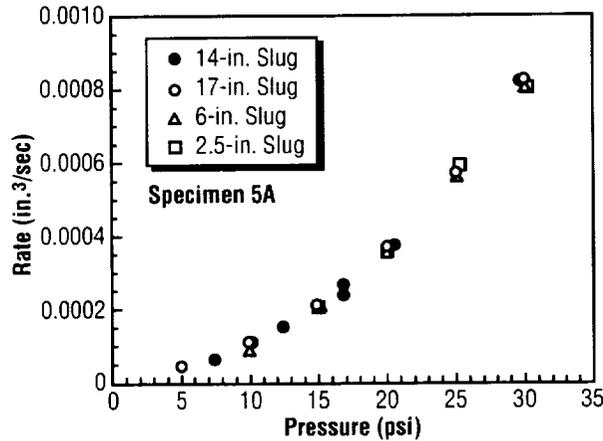


Figure 8. Flow rate using different lengths of liquid slugs for specimen 5A.

2.4.2.4 Size of Glass Tube. Three different sizes of glass tubes were used having inner diameters of 0.4, 1.2, and 3 mm. For a given flow rate, the slug will obviously travel slower in the larger diameter tube and more rapidly in the smaller diameter tube. Since the location of the slug at various times needs to be measured, the slug cannot be travelling too fast or accurate readings cannot be achieved. Also, if the flow rate is small, it can take a very long time to get a reading, unnecessarily slowing the data acquisition process. To examine the affect of tube size on flow rate, three tube sizes were used to measure the flow rate on specimen 5A. The results are presented in figure 9. Since a difference is clearly noted for the 0.4-mm tube, this test was performed on specimen 6B. The data are given in figure 10. Again the data show a much lower measured flow rate for the 0.4-mm tube, whereas the 3-mm and 1.2-mm tubes give excellent agreement.

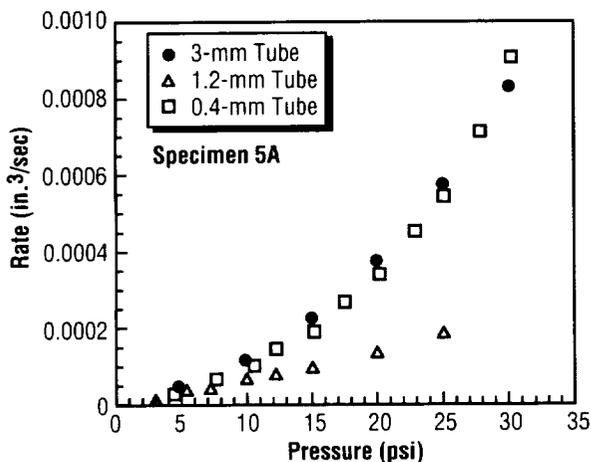


Figure 9. Flow rate measured on specimen 5A using different size tubes.

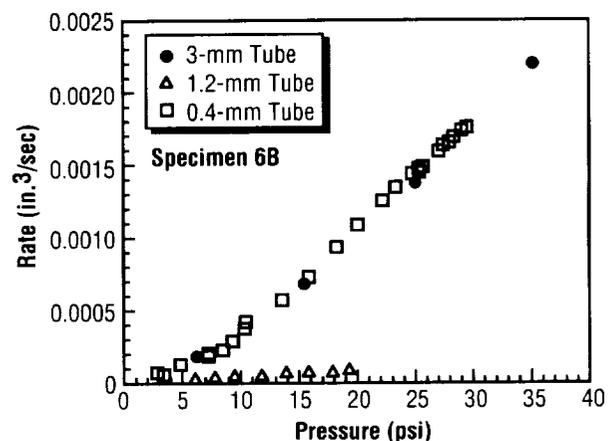


Figure 10. Flow rate measured on specimen 6B using different size tubes.

Using actual data, the pressure needed to overcome the viscous forces of the slug can be found from equation (5). The results for the three tube sizes used are shown graphically in figure 11 as a function of flow rate v . A slug length of 10 in. was assumed. The liquid was assumed to be isopropyl alcohol at room temperature. This plot shows just how critical the size of the tube can be when it comes to viscous forces. Not only does the smaller tube give rise to much higher viscous forces for a given flow rate, but for a given specimen the flow rate will be highest in the smallest tube, further increasing the viscous forces that need to be overcome.

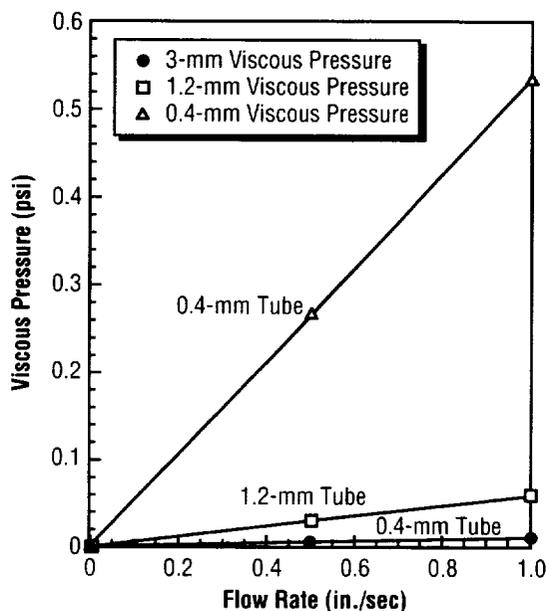


Figure 11. Plot of viscous pressure on slug as a function of flow rate.

From the ideal gas law

$$p = \frac{nRT}{V} \quad (6)$$

where

- p = pressure in the system
- n = number of moles of gas in the system
- R = universal gas constant
- T = absolute temperature
- V = volume of system.

At the start of the test, $p = 14.7$ psi (plus a small amount to keep the slug from falling back down the tube) and V is ≈ 1 in.³. Using these values as the starting point for the tests, the original number of moles of gas in the system turns out to be $\approx 6.8185 \times 10^{-4}$ moles. As more moles of gas are added (as the helium leaks in through the specimen), the volume of the system expands and/or the pressure increases. For a given flow rate, the increase in volume is given by

$$v_i = ft\pi r^2 , \quad (7)$$

where

- v_i = increase in volume
- f = flow rate (in./sec)
- t = time of flow (sec)
- r = radius of tube (in.).

The increase in the number of moles of gas in the volume is given by:

$$n_i = \frac{f}{1,367 \text{ in.}^3 / \text{mol}} , \quad (8)$$

where

- n_i = increase in moles of gas in the volume
- f = flow rate (in./sec)
- 1,367 in.³/mol = volume of one mole of an ideal gas.

Thus the increase in pressure is given by equation (6) with the new volume and number of moles added in

$$p_i = (6.8185 \times 10^{-4} \text{ mol} + n_i) \frac{\left(73.58 \frac{\text{in.} \cdot \text{lb}}{\text{mol} \cdot \text{K}} \right) (293 \text{ K})}{\left(1 \text{ in.}^3 + v_i \right)} - 14.7 \text{ psi} . \quad (9)$$

A plot of increase in pressure as a function of flow rate for the three tube sizes is given in figure 12. This was based on an elapsed test time of 60 sec and assumes the beginning volume was 1 in.³.

As mentioned earlier, the pressure generated by the increase in the number of moles of helium entering the volume must be greater than the viscous pressures or error will be introduced. As a check, data from figures 11 and 12 are superimposed for each size tube and presented in figures 13–15. The increase in pressure due to the added helium is termed the “driving” pressure and the pressure due to viscous forces is termed the “retarding” pressure.

In figure 13, it can be seen that at any flow rate the driving pressure will always be much greater than the viscous pressures, so very little error is introduced if the pressures due to slug viscosity are ignored. In figure 14, the driving pressure is higher than the viscous pressure, so little error should be measured when using this size tube; although, the difference here is not as great as that for the 3-mm tube. Figure 15 shows that the viscous pressures are always much larger than the driving pressures, thus error will be introduced into the flow rate measurements (they will be low). This is clearly demonstrated in the data from figures 9 and 10. Thus the 0.4-mm tube cannot be used with the specified parameters in this study.

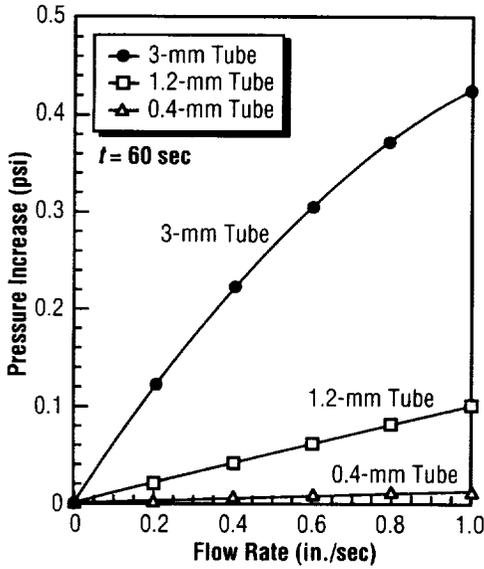


Figure 12. Increase in pressure as a function of flow rate.

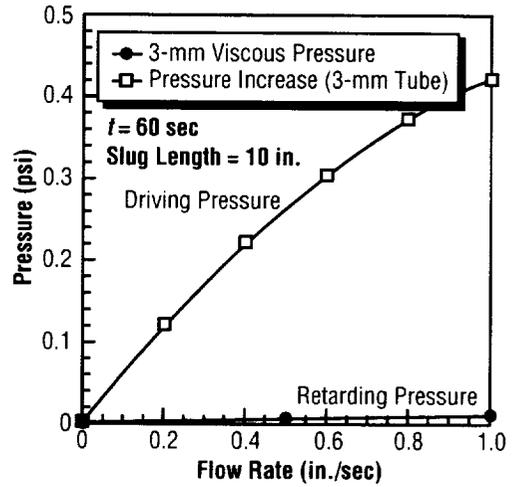


Figure 13. Comparison of driving and retarding (viscous) pressures for a 3-mm tube.

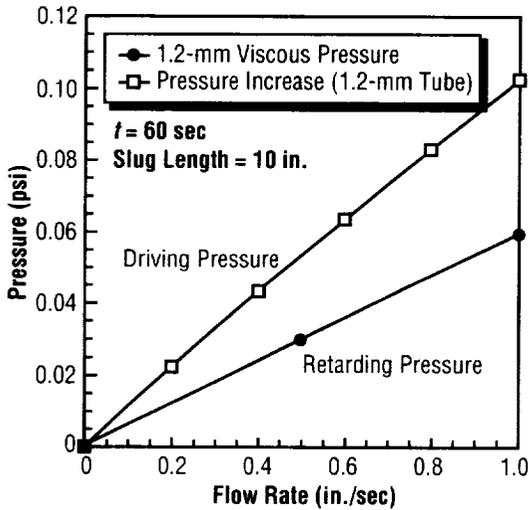


Figure 14. Comparison of driving and retarding (viscous) pressures for a 1.2-mm tube.

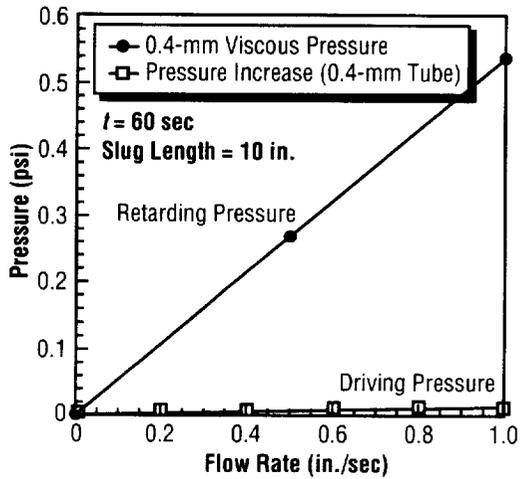


Figure 15. Comparison of driving and retarding (viscous) pressures for a 0.4-mm tube.

3. RESULTS

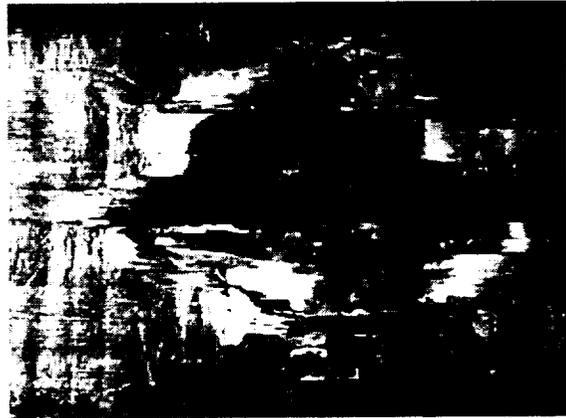
This section will present results of the impact, leak check, and permeability testing.

3.1 Impact Testing

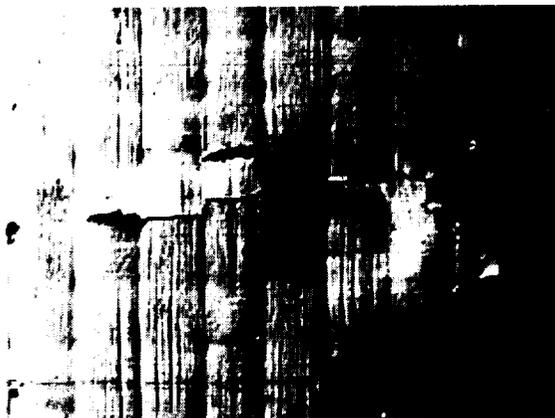
A total of 10 specimens was impacted, 2 at each of five energy levels. The resulting visual surface damage is presented in figure 16. Duplicates are not presented since the visual damage was nearly identical in every case. Damage of some form can be noted on all specimens, even those impacted at the smallest impact energy of 0.84 ft-lb.



(a) Front, specimen 2, 2.51 ft-lb



(b) Back, specimen 2, 2.51 ft-lb

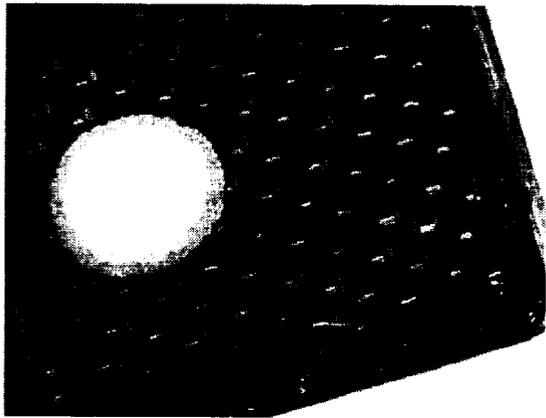


(c) Front, specimen 3, 2.09 ft-lb

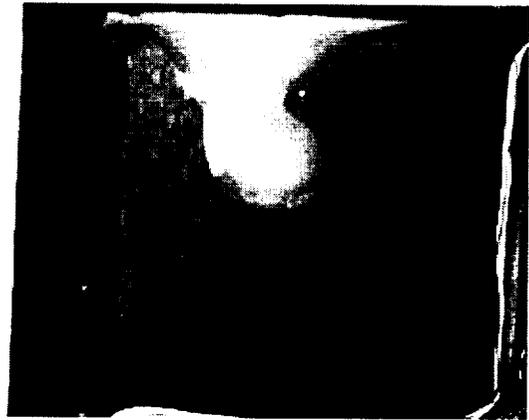


(d) Back, specimen 3, 2.09 ft-lb

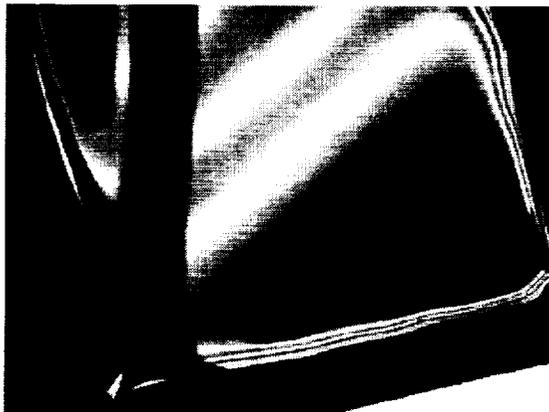
Figure 16. Surface views of impacted specimens.



(g) Specimen 5A, 1.26 ft-lb



(h) Specimen 5B, 1.26 ft-lb



(i) Specimen 6A, 0.84 ft-lb



(j) Specimen 6B, 0.84 ft-lb

Figure 17. Leak check images (continued).

All samples, with the exception of 6A, showed leakage. The larger the bubbles are in the pictures, the higher the leak rate. Specimen 2B was difficult to photograph due to the extremely large bubbles that were forming. Specimen 2A can be seen to rapidly expel the leak detect solution and form relatively large bubbles. As the impact damage becomes less severe, the bubbles become smaller. In fact, in specimens 5A and 6B, the leak rate is such that the leak detection fluid forms a fine “foam” that emanates from the impacted area.

3.3 Permeability Testing

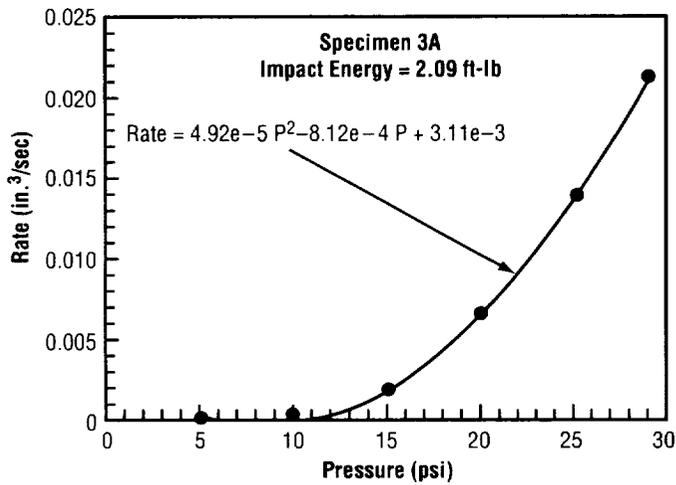


Figure 18. Flow rate (permeability) versus applied pressure for specimen 3A.

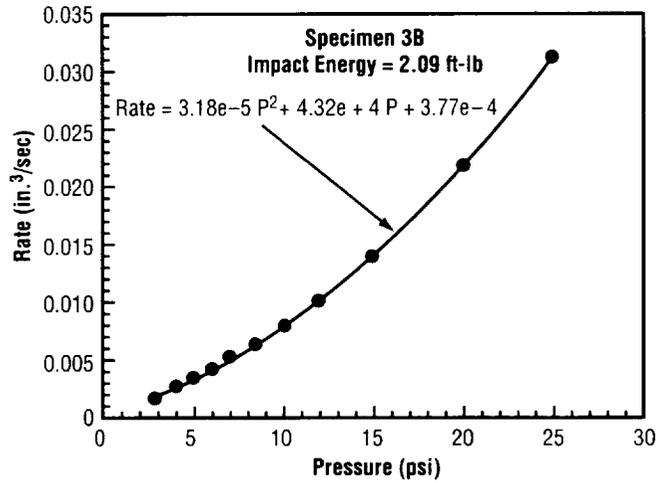


Figure 19. Flow rate (permeability) versus applied pressure for specimen 3B.

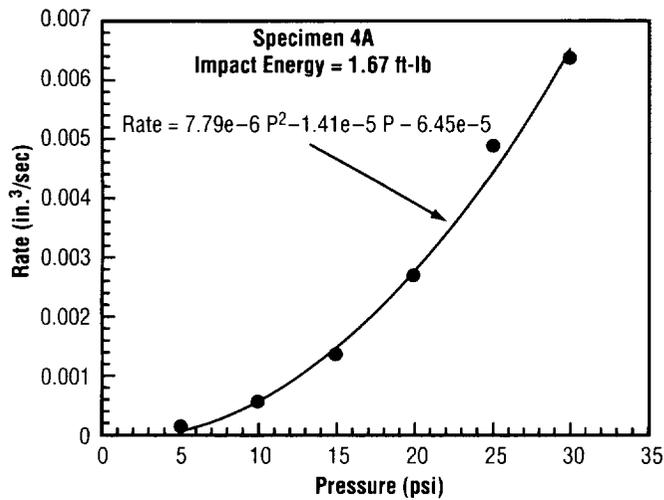


Figure 20. Flow rate (permeability) versus applied pressure for specimen 4A.

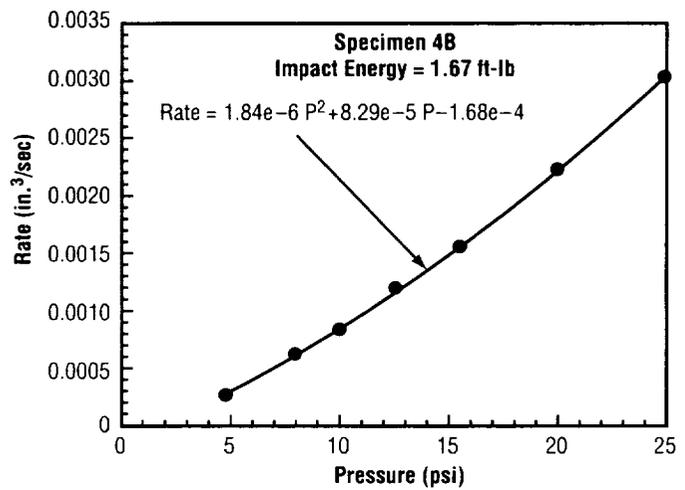


Figure 21. Flow rate (permeability) versus applied pressure for specimen 4B.

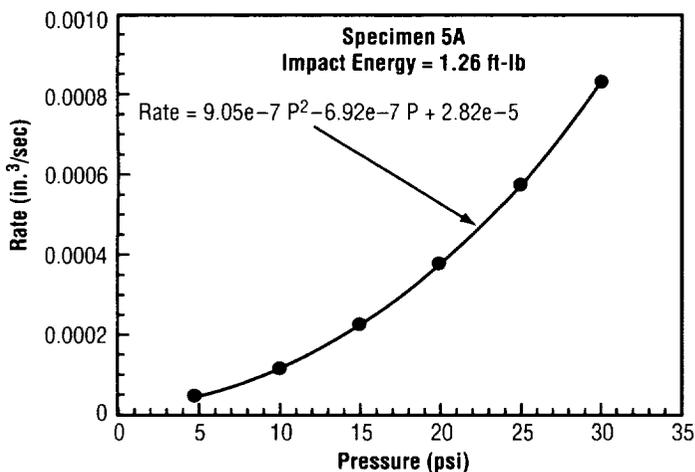


Figure 22. Flow rate (permeability) versus applied pressure for specimen 5A.

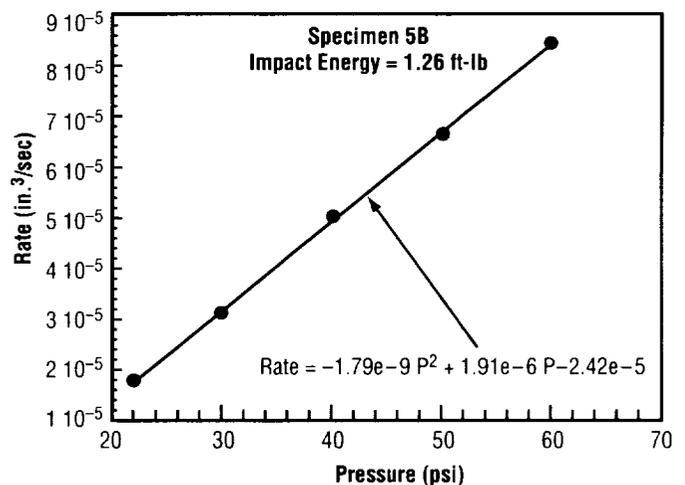


Figure 23. Flow rate (permeability) versus applied pressure for specimen 5B.

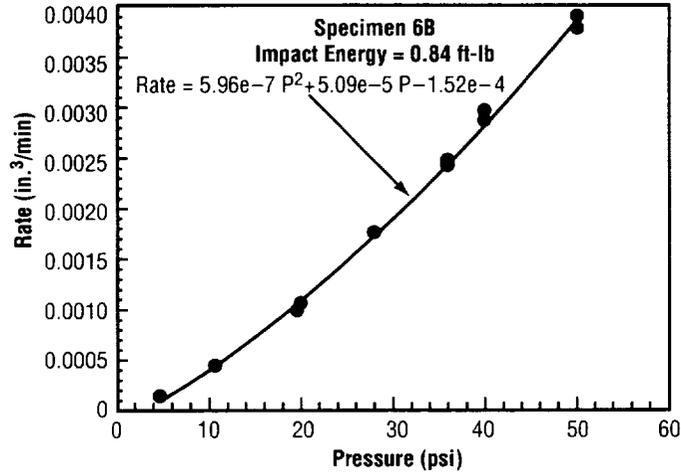


Figure 24. Flow rate (permeability) versus applied pressure for specimen 6B.

In general, the larger the impact energy the higher the flow rate for a given applied pressure, which is expected. A noticeable exception is specimen 5B, which showed a very low flow rate, even though it was hit harder than specimen 6B. The amount of nonlinearity in flow rate versus applied pressure also varied between samples; however, most of the nonlinearity is observed at the lower pressures and as the applied pressure increased, the linearity of the flow rate versus pressure increased.

4. CONCLUSIONS

A simple test apparatus can be used to measure permeability of impact damaged composite specimens, as long as the flow rate through the sample is not too great. Using the apparatus presented in this study, it was demonstrated that tube size is critical in obtaining proper flow rate readings. Too small of a tube can give rise to unacceptable levels of viscous forces on the liquid “slug” inside the tube, resulting in flow rate measurements that are too small. The type of liquid, length of the slug, and angle that the tube makes with the horizontal were all shown to have little or no affect on the flow rate readings.

For permeability-after-impact testing, the residual flow rate usually has a nonlinear dependence on the applied pressure, increasing more rapidly as a higher pressure is applied. Thus the permeability cannot be stated as a constant per unit of applied pressure.

The four-ply laminates tested in this study showed leakage after impact, even when visible damage could only be detected with magnifying techniques.

The qualitative measurement of leakage with the bubble-type leak detector solution corresponded with the qualitative permeability measurements.

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